

SYNTHESIS OF SUPERABSORBENT  
CARBONACEOUS FIBRE POLYMER VIA  
INVERSE-SUSPENSION POLYMERIZATION  
FOR NPK COATING

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I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Master of Science.

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## **STUDENT'S DECLARATION**

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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## ABSTRAK

Kebelakangan ini, penggunaan baja pelepasan terkawal (CRF) sebagai baja alternatif semakin meluas dalam industri pertanian. Baja ini merupakan baja yang mampu mengawal pelepasan nutrisi ke dalam tanah secara terkawal dan mampu memberi manfaat terhadap proses tumbesaran tumbuhan. Selain itu, CRF yang digabung dengan hydrogel mempunyai daya pengekal air dalam tanah yang tinggi serta kebolehan untuk melepaskan nutrisi ke dalam tanah secara perlahan dan terkawal. Kajian ini bertujuan menghasilkan Nitrogen-Fosforus-Kalium (NPK) bersalut polimer serat carbon superabsorbent (SPC) sebagai baja pelepasan terkawal. Bagi mencapai objektif utama, kajian ini dikelaskan kepada 3 peringkat berlainan; penghasilan serat karbon dari serat Kenaf melalui proses karbonisasi hidroterma (HTC), sintesis polimer superabsorbent (SAP) dan polimer serat karbon superabsorbent (SPC) menggunakan proses pempolimeran ampaian-songsang dan penghasilan baja lepasan terkawal; NPK bersalut SAP dan SPC. Pada mulanya, keluaran serat karbon dari serat Kenaf melalui proses HTC pada masa operasi yang berbeza telah dikaji. Penganalisis unsur telah digunakan bagi melihat keluaran serat karbon selepas proses HTC dijalankan dan semakin lama masa operasi, semakin meningkat jumlah keluaran serat karbon. Pencirian kumpulan berfungsi serat karbon diperoleh melalui FTIR dan analisis permukaan serat karbon diperhatikan melalui SEM. Kemudian, polimer serat karbon superabsorbent (SPC) dengan peratusan pengisi karbon berbeza (0.01wt%, 0.02wt%, 0.03wt%, 0.04wt% dan 0.05wt%) dan polimer superabsorben (SAP) telah disintesis. Sampel SAP bertindak sebagai sampel terkawal. Keupayaan penyerapan air SAP dan SPC disiasat menggunakan kaedah beg-teh. Sampel SPC dengan 0.04wt% mempunyai keupayaan optimum penyerapan air (55.279 g sampel air / g). Pencirian SAP dan SPC dilihat melalui analisis FTIR, FESEM dan EDX. Seterusnya, NPK bersalut SAP dan SPC dihasilkan dan pengekal air tanah dikaji selama 30 hari pada dua parameter; bahan salutan yang berlainan dan NPK bersalut dengan SPC dengan peratusan berat yang berbeza. NPK bersalut SAP dan SPC terbukti mempunyai keupayaan pengekal air yang tinggi dalam tanah berbanding NPK biasa. Pengekal air dalam tanah bagi sample NPK bersalut SPC merupakan yang terbaik berbanding sample lain kerana lebih 80% air masih kekal selepas 30 hari. Selain itu, kebolehan pengekal air bagi sample NPK bersalut SPC semakin meningkat selari dengan peningkatan peratusan berat baja tersebut di dalam tanah. Kadar pelepasan nutrisi bagi semua sampel CRF telah diperhatikan menggunakan spektrometri ICP-MS. Selama 30 hari, kesemua sampel menunjukkan kadar pelepasan nutrisi yang rendah dan terkawal dengan pelepasan nutrient N, P dan K kurang dari 50% berbanding sampel NPK. Manakala, sampel NPK bersalut SPC mempunyai kadar pelepasan nutrisi yang terendah berbanding sampel-sampel lain. Kinetik bagi mekanisme pelepasan nutrisi bagi sampel-sampel CRF dikaji berdasarkan Model Kosmeyer-Peppas. Mekanisma pelepasan bagi kedua-dua sampel menghampiri ciri-ciri pembauran Fickian kerana nilai  $n$  bagi kedua-dua sampel adalah kurang dari 0.5.

## ABSTRACT

Recently, the use of controlled-release fertiliser (CRFs) in agriculture has resulted in huge benefits in plant growth and cultivation. CRF is a type of modified fertiliser where a conventional fertiliser is conjoining with various materials, mostly polymers, creating a fertiliser with the ability to release nutrients in controlled manner. A superabsorbent polymer-coated fertiliser has the advantage in retaining water in soil after irrigation and also releasing nutrients into the soil slowly and under control. This study aimed to produce a nitrogen-phosphorus-potassium (NPK) fertiliser coated with superabsorbent carbonaceous fibre polymer (SPC) that possesses water-retention and controlled-release properties. In order to achieve the main objective, this study was divided into three different stages: producing carbonaceous fibre from natural kenaf fibre through a hydrothermal carbonisation (HTC) process; synthesising superabsorbent polymer (SAP) and superabsorbent carbonaceous fibre polymer (SPC) via inverse-suspension polymerisation; and lastly, producing and investigating the controlled-release and water-retention properties of NPK coated with SAP/SPC (the CRF). Firstly, the production of carbonaceous fibre from kenaf through HTC over different operating times was examined. An elemental analyser was used to measure the carbon content after HTC and indicated that carbon yield increases with time, with 12 h as the optimal operating time to produce the highest yield: 64.47%. Characterisation of the carbonaceous fibre was made with FTIR and SEM. Then, the SPC polymers with different amounts of filler (0.01, 0.02, 0.03, 0.04 and 0.05 wt %) were synthesised with SAP as control. The water absorbency ability of SAP and SPC were investigated using the tea bag method. SPC samples with 0.04 wt % had the optimal water absorbency ability (55.279 g water/g sample). Characterisation of SAP and SPC were observed through FTIR, FESEM and EDX analyses. Later, NPK fertilisers coated with SAP or SPC were produced and investigation of water retention in soil was made for 30 days, varying two different parameters: different coating material and different weight percentage of NPK coated with SPC. NPK coated with SAP or SPC proved to have high water-retention ability in soil compared to uncoated NPK. NPK coated with SPC showed better results than NPK coated with SAP, with more than 80% water retained after 30 days of experiment. In addition, the higher the weight percentage of NPK coated with SPC in the soil, the greater its water retention. The slow-release behaviour of all of the CRF samples were observed using ICP-MS spectrometry. Over 30 days, all samples of NPK coated with SAP/SPC had low release rates, with less than 50% nutrient release compared to uncoated NPK. NPK coated with SPC showed the lowest release rate. The release mechanism kinetics of NPK coated with SAP/SPC approached Fickian diffusion-controlled release as the  $n$  value for both samples were less than 0.5. In conclusion, Carbonaceous fibre from Kenaf was successfully incorporated into the SPC, improving the polymer performance in terms of water absorbency, water retention in soil and its controlled release mechanisms.

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## LIST OF SYMBOLS

$r^2$	Determination coefficient
D	Diffusion coefficient
$m_2$	Weight of Tea Bag After Reaching Equilibrium
$m_1$	Initial Weight of Tea Bag Before Immersion in Water
$m_i$	Daily weight (sample+soil)
$m_0$	Initial Weight (sample+soil+water)
t	Time System Release
k	Rate Constant
n	Release Exponent

## **LIST OF ABBREVIATIONS**

MPOB	Malaysia Palm Oil Board
LKTN	National Kenaf and Tobacco Board
HTC	Hydrothermal Carbonization Process
SAP	Superabsorbent Polymer
SPC	Superabsorbent Carbonaceous Polymer
CRF	Controlled Release Fertilizer
NPK	Nitrogen-Phosphorus-Potassium
ICP-MS	Induced Coupled Plasma- Mass Spectrometry
FTIR	Fourier Transform Infrared Spectroscopy
SEM	Scanning Electron Microscope
TGA	Thermogravimetric Analysis
NNMBA	N’N-Methylenebisacrylamide
APS	Ammonium Persulfate

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